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स्टाइरीन (विनाइल बेंज़ीन) — विशिष्टि  
( तीसरा पुनरीक्षण )

Styrene (Vinyl Benzene) —  
Specification  
( Third Revision )

ICS 71.080.15

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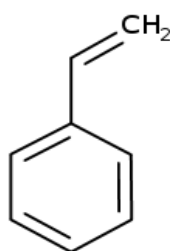
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## FORWORD

This Indian (Third Revision) Standard was adopted by the Bureau of Indian Standards after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Styrene ( $C_6H_5-CH=CH_2$ ) is used for the manufacture of styrene-butadiene rubber (SBR) polystyrene and for copolymerization products with other monomers to obtain synthetic resin binders and adhesives. It is also used in the packaging industry and in the production of toys, housewares and appliances. Styrene-divinyl benzene copolymer is used in the preparation of both anion and cation exchange resins. It is also known as phenyl ethylene or vinyl benzene. It is represented by the following structural formula:



STYRENE (VINYL BENZENE)

Molecular Mass: 104.15

The material undergoes polymerization during shipment or in storage due to the catalytic effect of heat and light. The commercial material is, therefore, stabilized with sulphur or other inhibitors like p-tertiary butyl catechol in small concentrations (*see* IS 14631 'Styrene — Code of safety')

This standard was first published in 1967 and subsequently revised in 1988 and 2020. First revision was done in view of the improved quality of the material being made available in the country. In the first revision, the requirement of refractive index and relative density have been included, limits of impurity stipulated and the methods of test for colour and aldehyde content modified. The second revision was undertaken mainly to update cross referred standards.

In this (*third*) revision, grade 2 has been deleted as mostly only one grade is manufactured across the world and the requirements of styrene of the existing grade is modified to 99.7, percent by mass. The test method for determination of colour, assay and sulphur have been modified. Further, alternate test method for determination of relative density, refractive index, freezing point, aldehyde, chloride, inhibitor content, polymer content and peroxides have been incorporated. Benzene being carcinogenic in nature and found as an impurity in styrene, the committee decided to incorporate benzene as characteristic with limit of 1 ppm. Requirement of solubility of polymer has been deleted as it is already calculated in the form of impurities.

(Continued on third cover)

*Indian Standard*  
**STYRENE (VINYL BENZENE) — SPECIFICATION**  
*( Third Revision )*

## 1 SCOPE

This standard prescribes the requirements, the methods of sampling and testing for the material commercially known as styrene (vinyl benzene).

## 2 REFERENCES

The standards listed in Annex A contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed in Annex A.

## 3 REQUIREMENTS

### 3.1 Description

The material shall be clear liquid free from sediment, suspended matter and undissolved water at 27 °C.

3.2 The material shall also comply with the requirements given in Table 1.

## 4 PACKING AND MARKING

### 4.1 Packing

4.1.1 Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in internally epoxy coated steel drums painted white externally.

4.1.2 All containers shall be dry, clean and free from substances soluble in styrene and leak-proof.

4.1.3 The containers shall be securely closed, protected from light and heat and shall be stored in a cool place.

4.1.4 All containers for storage and transport of the material, since classified as flammable and dangerous goods, shall in addition, comply with the requirements of the latest issue of Red Tariff and the requirements as laid down from time to time by the

Chief Inspector of Explosives, Government of India, for packing, storage and transit of flammable liquids and the Board of Trade Regulations as applicable thereon for transport by steamers (*see* IS 14631).

4.1.5 Except when they are opened for the purpose of cleaning and rendering them free from styrene vapour, all empty tanks or other containers shall be kept securely closed unless they have been thoroughly cleaned and freed from styrene vapour.

### 4.2 Marking

4.2.1 Each container shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of manufacturer and his recognized trade-mark, if any;
- c) Month and year of manufacture;
- d) Lot or batch number;
- e) Inhibitor content;
- f) Net weight; and
- g) Any other statutory requirements.

4.2.2 Each container shall have a caution label 'FLAMMABLE' together with the corresponding symbol for labelling of dangerous goods in accordance to IS 1260 (Part 1).

NOTE — Necessary safeguard against the risk arising from storage and handling of large volumes of flammable liquids shall be provided and all due precautions shall be taken at all times to prevent accidents by fire or explosion.

### 4.2.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

## 5 SAMPLING

The procedure for sampling and criteria for conformity of the material shall be as prescribed in Annex N.

**6 TEST METHODS**

**6.1** Test shall be conducted as prescribed in col (4) and col (5) of Table 1.

**6.2 Quality of Reagents**

Unless specified otherwise, pure chemicals and

distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

**Table 1 Requirements for Styrene (Vinyl Benzene)**

(Clauses 3.2, 6.1 and N-6.1.2)

SI No.	Characteristic	Requirement	Method of Test Ref to	
			Indian Standard	Clause No. of Annex
(1)	(2)	(3)	(4)	(5)
i)	Colour, Pt-Co, <i>Max</i>	15	IS 1448 (Part 178) <sup>1)</sup>	B
ii)	Relative density at 27 °C /27 °C	0.902 4 to 0.903 8	—	C
iii)	Refractive index at 27 °C, <i>Min</i>	1.542 6	IS 1448 (Part 158) <sup>1)</sup>	D
iv)	Freezing point <sup>2)</sup> , °C, <i>Min</i>	- 30.79	—	E
v)	Assay, percent by mass, <i>Min</i>	99.70	—	F
vi)	Aldehyde, percent by mass (as benzaldehyde), <i>Max</i>	0.02	—	G
vii)	Peroxides, percent by mass (as H <sub>2</sub> O <sub>2</sub> ), <i>Max</i>	0.01	—	H
viii)	Sulphur, percent by mass, <i>Max</i>	0.003	IS 1448 (Part 160) <sup>1)</sup>	J
ix)	Chlorides, percent by mass, <i>Max</i>	0.005	—	K
x)	Polymer content, ppm, <i>Max</i>	10	—	L
xii)	Inhibitor content (p-tertiary butyl catechol) <sup>3)</sup> , ppm	15-50	—	M
xiii)	Ethyl benzene, percent by mass, <i>Max</i>	0.05	—	F
xiv)	Alpha methyl styrene, percent by mass, <i>Max</i>	0.05	—	F
xv)	Benzene, ppm, <i>Max</i>	1	—	F

<sup>1)</sup> In case of disputes, IS 1448 (Part 178) shall be the referee method for determining colour; IS 1448 (Part 158) shall be the referee method for determining refractive index and IS 1448 (Part 160) shall be the referee method for determining sulphur content.

<sup>2)</sup> The freezing point is optional parameter to be tested.

<sup>3)</sup> Recommended for one month's safe storage under Indian climatic conditions. May be increased or decreased by mutual agreement between the purchaser and the supplier.

## ANNEX A

(Clause 2)

## LIST OF REFERRED STANDARDS

<i>IS No.</i>	<i>Title</i>	<i>IS No.</i>	<i>Title</i>
IS 170 : 2020	Acetone — specification ( <i>fifth revision</i> )		Fuels — Ultraviolet fluorescence method
IS 265 : 2021	Hydrochloric acid — Specification ( <i>fifth revision</i> )	(Part 178) : 2020/ISO 6271 : 2015	Part 178 Clear liquid — Estimation of colour by the platinum cobalt colour scale
IS 321 : 1964	Specification for absolute alcohol ( <i>first revision</i> )	IS 1997 : 2008/ISO 385 : 2005	Laboratory glassware — Burettes ( <i>third revision</i> )
IS 517 : 2020	Specification for methanol (methyl alcohol) ( <i>third revision</i> )	IS 2214 : 1977	Specification for silver nitrate, pure, and analytical reagent ( <i>first revision</i> )
IS 1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )	IS 2263 : 1979	Methods of preparation of indicator solutions ( <i>first revision</i> )
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods ( <i>first revision</i> )	IS 2303 (Part 2) : 2018/ISO 4802-1 : 2016	Grading glass for alkalinity: Part 2 Hydrolytic resistance of glass containers — Determination by titration method and classification ( <i>second revision</i> )
IS 1448	Methods of Test for Petroleum and its Products:		
(Part 158) : 2016/ISO 5661 : 1983	Part 158 Hydrocarbon liquids — Determination of refractive index	IS 4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures ( <i>first revision</i> )
(Part 160) : 2017	Part 160 Determination of sulphur content of automotive	IS 14631 : 1999	Styrene — Code of safety

## ANNEX B

[Table 1, Sl No. (i)]

## DETERMINATION OF COLOUR BY TRISTIMULUS COLORIMETER

## B-1 APPARATUS

## B-1.1 Tristimulus Colorimeter

## B-2 REAGENTS

B-2.1 Cobalt Chloride ( $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ )

**B-2.2 Concentrated Hydrochloric Acid** — specific gravity : 1.19

B-2.3 Potassium Chloroplatinate ( $\text{K}_2\text{PtCl}_6$ )

## B-2.4 Platinum-Cobalt Stock Solution

Dissolve 1.245 g of potassium chloroplatinate ( $\text{K}_2\text{PtCl}_6$ ) and 1.00 g of cobalt chloride ( $\text{CoCl}_2 \cdot \text{H}_2\text{O}$ ) in water. Add 100 ml of hydrochloric acid (HCl) and dilute it to 1 litre with distilled water. The absorbance measured for 500 platinum-cobalt stock solution in a cell having a 10 mm light path with distilled water in a matched cell as the reference solution, shall lie within the limits as given in Table 2.

**B-2.5 Platinum-Cobalt Standard Solution**

Prepare Pt-Co standard solutions as given in Table 3 from Pt-Co stock solution by diluting required volume to 100 ml with distilled water in volumetric flask. Pt-Co standard solution with higher values may be prepared by using proportionally greater volume of stock solution (*see* Table 4).

manual. Take the samples and measure the samples in a cell of the selected path length. Perform three measurements without replacing the sample. Calculate the average taken from the three measurements to be the Pt-Co reading for the sample.

**B-3 PROCEDURE**

Set the instrument as per instrument manufacturer's

**Table 2 Absorbance Tolerance Limits for number 500 Platinum-Cobalt Stock Solution**  
(Clause B-2.4)

Sl No.	Wavelength, nm	Absorbance
(1)	(2)	(3)
i)	430	0.110 to 0.120
ii)	455	0.130 to 0.145
iii)	480	0.105 to 0.120
iv)	510	0.055 to 0.065

**Table 3 Pt-Co Colour Standards**  
(Clause B-2.5)

Sl No.	Colour Standard Number	Stock Solution, ml	Colour Standard Number	Stock Solution, ml
(1)	(2)	(3)	(4)	(5)
i)	1	0.20	10	2.00
ii)	2	0.40	11	2.20
iii)	3	0.60	12	2.40
iv)	4	0.80	13	2.60
v)	5	1.00	14	2.80
vi)	6	1.20	15	3.00
vii)	7	1.40	20	4.00
viii)	8	1.60	25	5.00
ix)	9	1.80	30	6.00

**Table 4 Pt-Co Colour Standards Solution with Higher Values**  
(Clause B-2.5)

Sl No.	Colour Standard Number	Stock Solution, ml	Colour Standard Number	Stock Solution, ml
(1)	(2)	(3)	(4)	(5)
i)	5	1	70	14
ii)	10	2	100	20
iii)	15	3	150	30
iv)	20	4	200	40
v)	25	5	250	50
vi)	30	6	300	60
vii)	35	7	350	70
viii)	40	8	400	80

## ANNEX C

[Table 1, Sl No. (ii)]

## DETERMINATION OF RELATIVE DENSITY

## C-1 GENERAL

Two methods namely, Method A Determination of relative density by manual method and Method B Determination of relative density by digital density analyzer have been prescribed. In case of dispute, Method B shall be the referee method.

## C-2 METHOD A

## C-2.1 Outline of the Method

In this method, masses of equal volumes of the material and water at the same temperature are compared using relative density bottle.

## C-2.2 Apparatus

**C-2.2.1 Relative Density Bottle** — 25 ml capacity

**C-2.2.2 Water-Bath** — maintained at  $(27.0 \pm 0.2) ^\circ\text{C}$

**C-2.2.3 Thermometer** — any convenient thermometer of suitable range with 0.1 or 0.2 degree sub-divisions

## C-2.3 Procedure

Clean and dry the relative density bottle, weigh and then fill with recently boiled and cooled distilled water at  $27 ^\circ\text{C}$ . Fill to overflowing by holding the relative density bottle on its side in such a manner to prevent entrapment of air bubbles. Insert the stopper and immerse in a water-bath maintained at  $(27 \pm 0.2) ^\circ\text{C}$ . Keep the entire bulb completely covered with water and hold at that temperature for 30 min. Carefully, remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, cool and weigh. Calculate the mass of water. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

## C-2.4 Calculation

$$\text{Relative density at } 27/27 ^\circ\text{C} = \frac{A-B}{C-B}$$

where

$A$  = mass of the relative density bottle filled with the material, in g;

$B$  = mass of the clean and dry relative density bottle, in g; and

$C$  = mass of the relative density bottle filled with water, in g.

## C-3 METHOD B

## C-3.1 Outline of the Method

In this method, the digital density meter used in conducting a test measures the density accurately by electronically measuring the period of oscillation and compensating for viscosity.

## C-3.2 Apparatus

**C-3.2.1 Digital Density Analyzer**

**C-3.2.2 Circulating Constant-Temperature Bath**

**C-3.2.3 Syringes (for manual injection)** — at least 2 ml

**C-3.2.4 Auto Sampler (automated injection analyses)**

**C-3.2.5 Temperature Sensing Device (TSD)**

**C-3.2.6 Ultrasonic Bath** — unheated (optional), of suitable dimensions to hold container(s) placed inside of bath, for use in effectively dissipating and removing air or gas bubbles that may be entrained in viscous sample types prior to analysis

## C-3.3 Procedure

## C-3.3.1 For Manual Injection

Add small amount of sample, about 1 ml to 2 ml into the clean, dry sample tube of the instrument using a syringe. Make sure the sample tube is completely filled and is free of air bubbles. The sample must be completely homogenous and free of any gas bubbles. Optical or physical methods can be used to check the integrity of the filled sample for the presence of gas bubbles. Empty and refill the sample tube if gas bubbles are identified, then recheck for gas bubbles.

Record the relative density once the instrument displays a stable reading to four significant figures for relative density or five for T-value, indicating that thermal equilibrium has been attained.

## C-3.3.2 For Automated Injection

When evaluating samples via automatic injection, an auto sampler is used. The manufacturer's instructions are followed for assuring the test specimen's integrity before and transferring

a representative test specimen into the instrument for analysis. When using an auto sampler, a single determination is sufficient.

Record the relative density once the instrument displays a stable reading to four significant figures for relative density, determined by the analyses as appropriate.

### C-3.4 Calculation

#### C-3.4.1 Calculating density analyzers

The recorded value is the result.

#### C-3.4.2 Non-Calculating Density Analyzers

Instrument constants ( $K_2$ ) =  $(1.0\,000 - d_a)/[(T_w)^2 - (T_a)^2]$

where

$T_a$  = observed period of oscillation for cell containing air, in  $\mu$ s;

$T_w$  = observed period of oscillation for cell containing water, in  $\mu$ s; and

$d_a$  = density of air at test temperature, in g/ml.

Density of air ( $d_a$ , g/ml) =

$$0.001\,293\,(273.15/T)\,(P/101.325)$$

where

$T$  = temperature, K; and

$P$  = barometric pressure, kPa

Calculate the density or relative density by following formulae, using the observed  $T$ -value for the sample and the  $T$ -value for water, as well as the relevant instrument constants:

$$\text{Relative density} = 1 + K_2 (T_s^2 - T_w^2)$$

where

$K_2$  = instrument constant for relative density;

$T_s$  = observed period of oscillation for cell containing sample; and

$T_w$  = observed period of oscillation for cell containing water.

All calculations should be done to six significant numbers, and the final results should be rounded to four.

NOTE — Any other method similar to digital density meter may also be used.

## ANNEX D

[Table 1, Sl No. (iii)]

### DETERMINATION OF REFRACTIVE INDEX

#### D-1 GENERAL

Three methods namely, Method A Determination of refractive index by refractometer and Method B Determination of relative density by automatic digital refractometer and IS 1448 (Part 158) have been prescribed. In case of dispute, IS 1448 (Part 158) shall be the referee method.

#### D-2 METHOD A

##### D-2.1 General

To this determination, the refractive index of a material is taken as the ratio of the sine of the angle of incidence to the sine of the angle of refraction when a ray of light of wavelength 589 Å (the mean of the D lines of sodium) passes from air into the material.

The notation is refractive index  $n_D^t$ ,  $t$  is the temperature (°C) at which the determination is made.

##### D-2.2 Procedure

Determine the refractive index in a standard instrument\* employing the principle of the critical angle, using diffused daylight or any convenient artificial light as illuminant. Maintain the prisms at the specified temperature and allow the material to stay on the prism surface for a couple of minutes to attain the required temperature. Take a second reading after the laps of a few minutes.

Since moisture in the air may condense on the cooled prisms, great care shall be exercised when determining refractive indices during hot, humid weather. Occasionally, the instrument should be checked by means of the quartz plate that accompanies it, using monobromonaphthalene, or if such a plate is not available, by means of distilled water, at 30 °C. The refractive index of distilled water at 30 °C is 1.332 200.



If, for any reason, the refractive index cannot be determined at the specified temperature, apply the correction factor of 0.000 38 per degree celsius if a factor is not so specified. If the refractive index is determined at a temperature above the specified temperature, add the appropriate correction, if determined below the specified temperature, subtract the appropriate correction.

Report the refractive index at 27 °C as a number correct to four decimal places.

\*A suitable type of instrument for this purpose is the Abbe Refractometer

## D-3 METHOD B

### D-3.1 Apparatus

**D-3.1.1 Refractometer** — automatic digital refractometer with a suitable measuring range of 1.330 0 to 1.500 0 or higher, capable of displaying the measured refractive index automatically and digitally. The instrument shall incorporate a solvent resistant prism seal.

#### D-3.1.2 Temperature Control Unit

**D-3.1.3 Temperature Measuring Device** — a platinum resistance probe or equivalent. The accuracy and resolution of the temperature measuring device shall be 0.1 °C.

**D-3.1.4 Light source** — filtered white light such as that obtained using a tungsten-halogen lamp or a light emitting diode (LED), capable of providing the sodium D spectral line at 589 nm.

#### D-3.1.5 Light Filters

### D-3.2 Reagents

**D-3.2.1 *n*-Pentane** (purity 95, percent, minimum) — used for cleaning the prism faces

**D-3.2.2 Toluene** — HPLC Grade, used for cleaning the prism faces

**D-3.2.3 1-Bromonaphthalene** (purity 98, percent minimum) — contact liquid when calibrating with

solid reference standard.

**D-3.2.4 Primary Reference Materials** — for calibrating the instrument

**D-3.2.4.1 Solid reference standard** — with the value of refractive index engraved on its upper face

**D-3.2.4.2 Distilled or deionized water** — at 20 °C,  $n_D = 1.333\ 0$ ; at 25 °C,  $n_D = 1.3325$ ; and at 30 °C,  $n_D = 1.331\ 9$

**D-3.2.4.3 2, 2, 4-trimethylpentane** (purity 99 percent, minimum), at 20 °C,  $n_D = 1.391\ 5$ ; at 25 °C,  $n_D = 1.389\ 0$

**D-3.2.4.4 Methylcyclohexane** (purity 99 percent, minimum) — at 20 °C,  $n_D = 1.423\ 1$ ; at 25 °C,  $n_D = 1.4206$

**D-3.2.4.5 Toluene** (purity 99 percent, minimum) — at 20 °C,  $n_D = 1.496\ 9$ ; at 25 °C,  $n_D = 1.494\ 1$ .

**D-3.2.5 Secondary Reference Materials** — for calibrating the instrument

**D-3.2.5.1 Mineral oil calibration standards** — measured and certified by suppliers for specific refractive index ranges and temperatures

### D-3.3 Procedure

The prism, the surrounding plate and dish/plate (and presser, if fitted) are thoroughly cleaned. Confirm that the prism temperature is correct, and the instrument is properly calibrated. The test sample is introduced to the prism, making sure that the prism is adequately covered. Do not introduce an excessive amount as this can slow the thermal equilibration process. The sample presser is lowered, if fitted.

A series of readings is noted, until a steady value is obtained. Once the steady state is obtained, report, the steady value as the refractive index of the sample at the test temperature.

## ANNEX E

[Table 1, Sl No. (iv)]

### DETERMINATION OF FREEZING POINT

#### E-1 GENERAL

Two method namely, Method A determination of freezing point in equilibrium with air atmospheric

pressure and Method B determination of freezing point by phase transition method. In case of disputes, method B shall be the referee method.

**E-2 METHOD A****E-2.1 Outline of the Method**

The freezing point of the material, which is a measure of its assay, is determined in equilibrium with air atmospheric pressure. The presence of small amounts of impurities causes depression of the freezing point which is proportional to the molar concentration of the contaminating substances. The freezing point is determined from the freezing curve, a graph of temperature versus time readings obtained when a stirred sample of the styrene is gradually cooled and frozen in a sample tube

surrounded by an air bath refrigerated with dry ice. The measurements are made directly on sample without preliminary purification as small amounts of polymer do not affect the results because of low molar concentration. Any water present is frozen out of the sample and does not affect the results. The method is applicable to samples in which the major impurity is ethyl benzene.

**E-2.2 Apparatus**

**E-2.2.1 Thermometer** — specially designed to confirm to the following specifications:

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Liquid	: Mercury
Filling above mercury	: Nitrogen gas
Range and subdivision	: - 33.02 °C to - 29.98 °C in 0.02 intervals with auxiliary scales from - 0.2 °C to + 0.2 °C
Total length	: 405 mm to 410 mm
Immersion	: Total
Stem	: Plain front, enamel back, suitable thermometer tubing, diameter 6 mm to 7 mm
Bulb	: of suitable thermometric glass; diameter 6 mm to 7 mm but not greater than that of stem, length 50 mm to 60 mm
Distance from bottom of bulb to -33 degree mark	: 130 mm to 135 mm
Distance from top of the thermometer to -30 degree mark	: 125 mm to 135 mm
Length of unchanged capillary between the enlargement and the graduation next below	: Not less than 15 mm
Length of unchanged capillary above or below the 0° point	: Not less than 15 mm
Expansion chambers	: To permit heating to at least 50 °C
Top finish	: Plain

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(Concluded)

Graduation	: All lines, figures and letters to be clear cut and distinct. The graduation marks to be fine, straight, of uniform width and perpendicular to the axis of the thermometer. Each degree and the tenth-degree line to be longer than the intermediate ones. Graduation to be numbered in full at each degree mark and in decimals at other multiples of 0.2°.
Special marking	: The manufactures name or trade-mark, a serial number and the words 'total immersion' shall be etched on the stem
Scale error tolerance	: The error at any point on the scale shall not exceed 0.1 °C

## NOTES

**1** The accuracy attainable with mercury-in-glass thermometers, based on performance, when all precautions are taken, for measurements of the kind for which these thermometers were designed, is from 0.01 °C to 0.03 °C, and calibration corrections are stated to the nearest 0.01. Subdivision in 0.02 intervals, therefore, serves principally for the purpose of facilitating readings. The thermometer shall bear a valid calibration certificate from any institution authorised to issue calibration certificate traceable to international or national measurement standards and periodically checked for its accuracy by ice point determination or against bromobenzene standard (*see E-2.7*) or both.

**2** Digital thermometer may also be used.

**E-2.2.2 Freezing Point Apparatus** — it shall be of the dimensions and shape as given in Fig. 1, made of glass conforming to IS 2303 (Part 2).

**E-2.2.3 Bath Thermometer** — for reading the temperature of the cryostat bath. It should read as low as - 50 °C and be graduated in 1° sub-divisions.

**E-2.3 Reagents**

**E-2.3.1 Freezing Mixture** — a mixture of equal parts by volume of acetone and carbon tetrachloride for filling the dewar flask.

**E-2.3.2 Carbon Dioxide** — solid (dry ice)

**E-2.3.3 Bromobenzene** — extra pure, this should be kept in a dark bottle away from direct sunlight

**E-2.4 Sample Size and Precautions for Storage**

A sample of 20 ml to 25 ml is sufficient for duplicate determination. Samples should be kept refrigerated or inhibited with a minimum of 10 ppm of *p*-tertiary butyl catechol to prevent polymerization.

**E-2.5 Procedure**

**E-2.5.1** Use a sample of approximately 10 ml so that the top of the thermometer bulb will be immersed 2 cm when the bottom of the bulb is placed 1 cm above the bottom of the sample tube. Fill the dewar flask with the acetone-carbon tetrachloride mixture previously cooled to about 42 °C (by the addition of dry ice) to a point at least 1 cm above the level of the styrene in the freezing cell. Maintain the bath temperature between - 44 °C and - 48 °C during the

determination, by cautiously adding dry ice, a small piece at a time. Make sure that the bath does not foam excessively and that the carbon dioxide vapour is not allowed to contaminate the sample.

**E-2.5.2** Adjust the stirrer to 100 to 150 strokes per min and stir continuously throughout the determination. Be sure that the coil of the stirrer reaches the bottom of the sample tube and does not emerge from the liquid while in operation. Record the temperature to the nearest 0.01 °C at 30 s intervals and plot the readings of temperature versus time on graph paper (temperature - time data may be plotted directly at the time of observation upon graph paper prepared in advance). The freezing point of the sample, uncorrected, is the point of intersection of the liquid cooling curve of the temperature versus time graph with the freezing portion of the graph extrapolated back over the region of super cooling.

**E-2.6 Calculation**

**E-2.6.1** The uncorrected freezing point as obtained by extrapolation (*see E-2.5.2*) must be corrected by adding to it, algebraically, the following quantities:

- The calibration correction obtained by comparison to the standard thermometer;
- The change in ice-point reading of the thermometer obtained by comparison to its initial calibration against the standard thermometer; and
- The correction for the emergent stem, which may always be taken as equal to - 0.02 °C.

**E-2.6.2 Precision**

Shall be 0.05 mass percent of styrene over the range of 95 percent to 100 percent from styrene. Freeze a portion of the standard assuming the only impurity to be ethyl benzene.

**E-2.7 Use of Bromobenzene for Standardization**

**E-2.7.1** The apparatus and procedure are to be checked with bromobenzene which has been a freezing point close to that of styrene, under the following conditions:

- a) When any doubt arises about the accuracy of results;
- b) Whenever a new thermometer is used;
- c) Whenever a new operator uses the apparatus; and
- d) At least once every month with experienced operators and old thermometers.

**E-2.7.2** Make sure that the thermometer, stirrer and the sample tube are clear and free from styrene. Freeze a portion of the standard sample of bromobenzene exactly as if running a test on styrene. Obtain the uncorrected freezing point by extrapolation of freezing curve and apply the thermometer corrections. The corrected freezing point should agree with the freezing of the sample originally determined with the standard thermometer within  $\pm 0.02$  °C.

If such agreement is not obtained, the ice-point of the thermometer should be determined as in **E-2.7.3**. If agreement between the corrected and predetermined freezing is still not obtained, a source of error, particularly contamination of the sample with carbon dioxide, should be looked for in the method being followed. If necessary, as a final resort, the thermometer shall be recalibrated.

**E-2.7.3 Determination of Ice-Point**

Wash pieces of clear ice and crush them to a size not greater than 5 mm. Make a thick slurry by mixing a small amount of cooled distilled water with the crushed ice. Use a clean scoop rather than the hands for the manipulation of ice and the ice and water mixture. Fill the vessel with this mixture, tapping it down thoroughly. If the vessel is unsilvered, wrap it with a cloth. Support the thermometer in the ice and water mixture so that the zero-degree mark is at the ice level (a convenient support is made by putting a one-hole rubber stopper on top of the thermometer and clamping it). Place not more than two thermometers in the bath at one time and be sure that

the thermometers are suspended closer to the centre than to the outside of the cylinder. Note the time of immersion in the bath. Read the thermometer about 5 min after immersion in the bath. Take this reading as the ice-point, keeping the eye level with the top of the mercury column when taking the observations. The thermometer may be withdrawn slightly for readings but do not expose the enlargement which is below the auxiliary scale.

**E-3 METHOD B****E-3.1 Outline of Method**

The freeze point of styrene is determined by automatic phase transition technique, in which the styrene is first cooled until the presence of hydrocarbon crystals are formed and detected by optical detectors, followed by warming the sample back to its liquid stage. The freeze point is determined when the last styrene crystals return back to the liquid phase again.

**E-3.2 Apparatus****E-3.2.1 Automatic Freeze Point Chamber**

**E-3.2.2 Optical Detectors** — to monitor the transparency of the sample

**E-3.2.3 Thermoelectrical Device** — capable of controlling a wide temperature range

**E-3.2.4 Temperature Sensor** — to provide temperatures readings to 0.1 °C

**E-3.3 Procedure**

Risen the sample cup twice by pipetting  $(0.15 \pm 0.01)$  ml of sample into the cup and then cleaning the sample out of the cup by using a cotton swab each time. Now take  $(0.15 \pm 0.01)$  ml of sample again into sample cup and close airtight with the test chamber lid. The operation of the apparatus is started according to the manufacturer's instructions. All operations are controlled automatically. The internal purge gas will flow through the chamber to purge out any moist air trapped in the chamber. The styrene liquid is cooled at a rate of 15 °C/min to 30 °C/min until hydrocarbon crystals are formed and the optical detectors monitor the formation of crystals, followed by warming at a rate of 10 °C/min  $\pm$  0.5 °C/min until the styrene is in the liquid phase again.

NOTE — If cross contamination is of significant concern, heptane may be used to rinse and thoroughly clean the sample cup.

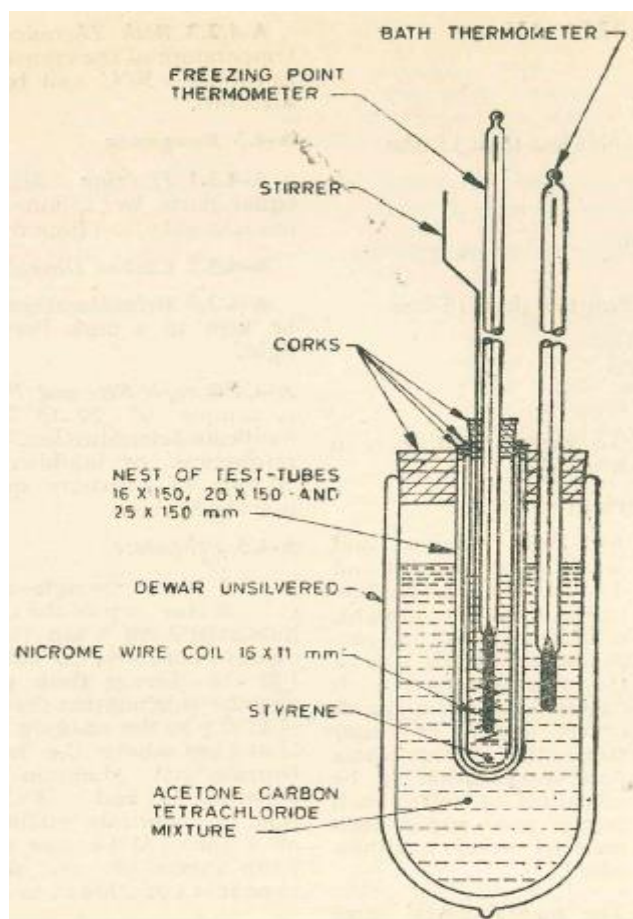


FIG. 1 FREEZING POINT APPARATUS

## ANNEX F

[Table 1, Sl No. (v), (xiii), (xiv) and (xv)]

**DETERMINATION OF STYRENE (VINYL BENZENE) CONTENT (ASSAY) AND ITS IMPURITIES BY GAS CHROMATOGRAPHY****F-1 OUTLINE OF THE METHOD**

A sample of the material is injected into the gas chromatograph from where it is carried by the carrier gas from one end of the column to the other end. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after the other and are detected by suitable means whose response is related to the

amount of specific component leaving the column.

**F-2 APPARATUS**

Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. A typical chromatogram using such a chromatograph as shown in Fig. 2 which may be obtained with the following chromatographic conditions:

---

Column	: Copper column with length: 1 100 mm and internal diameter: 4 mm, filled with 3 percent apiezon L with 0.25 percent tertiary butyl catechol on 30-60 mesh particle size.
Carrier gas	: Hydrogen
Detector	: Thermal conductivity
Conditions	: Column temperature, initially 170 °C and temperature programming at 5 °C/min from start. Injection temperature: 150 °C Detector temperature: 220 °C Flow rate, 45.0 ml/min
Sample size	: 2 µl

---

## NOTES

1 The above gas chromatographic conditions are suggestive. However, any GC having different columns (packed/Capillary having different length/diameter/film thickness) and different carrier gas (He, H<sub>2</sub> or N<sub>2</sub>), with different calibration technique (internal standard, external standard, area normalization) may be used provided standardization/ calibrations are done after setting up chromatographic conditions for required resolution.

2 Variations in conditions to arrive at the typical chromatographic separation shown in Fig. 2 are possible.

**F-3 PROCEDURE**

Conduct the flow of the carrier gas and inject sample at injection port where it is vaporized and well mixed with carrier gas. This is led into the chromatographic column wherein vapourized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. For this separation to be efficient, it is necessary that the column is maintained at the

temperature suggested throughout the time required for the resolution of the constituents. As the sample enters the detector, it gives the single corresponding to the amount of particular constituents leaving the column. The detector signal, on transmission to the recorder, plots the chart. From the areas under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

#### F-4 CALCULATION

**F-4.1** Calculate the peak areas of individual constituents pertaining to ethyl benzene and alphas-methylstyrene on the chromatogram of the material. The concentration of these impurities may be obtained on the basis of peak area on

chromatogram obtained with known amount of pure ethyl benzene and alphas-methylstyrene using the same apparatus under identical conditions.

**F-4.2** Assay, percent by mass, is determined by subtracting the sum of percent impurities from 100.

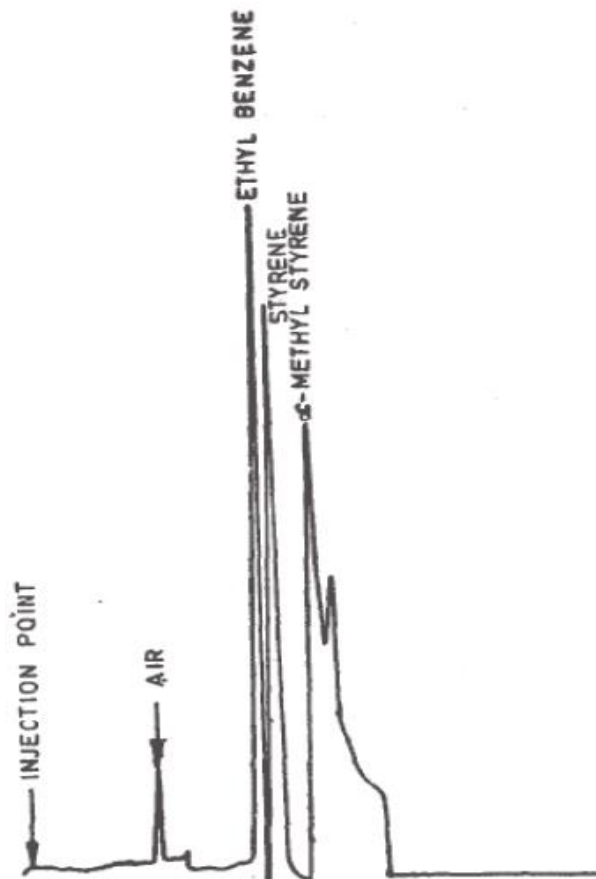


FIG. 2 A TYPICAL CHROMATOGRAM FOR STYRENE

#### ANNEX G

[Table 1, Sl No. (vi)]

#### DETERMINATION OF ALDEHYDE CONTENT

##### G-1 GENERAL

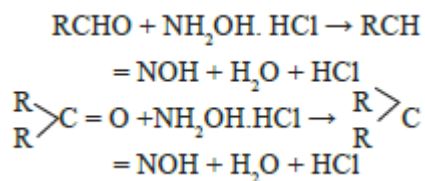
Two methods namely, Method A determination of aldehyde content by potentiometric titration and Method B determination of aldehyde content by UV spectrophotometry have been prescribed. In case of disputes, method B shall be the referee method.

##### G-2 METHOD A

###### G-2.1 Outline of the Method

A sample of styrene is added to an alcoholic solution of hydroxylamine hydrochloride. The aldehydes react with hydroxylamine hydrochloride any

ketones present will be partially included according to the following equation:



The hydrochloric acid liberated, which is equivalent to the aldehyde present in the sample, is titrated with standard alkali.

## G-2.2 Reagents

### G-2.2.1 Hydroxylamine Hydrochloride Solution

Dissolve 20 g of hydroxylamine hydrochloride (reagent grade) in 1 l of methanol and neutralize to the red-yellow end-point of thymol blue indicator.

**G-2.2.2 Standard Sodium Hydroxide Solution** — 0.05 N

**G-2.2.3 Hydrochloric Acid** — 0.05 N

**G-2.2.4 Methanol** (see IS 517)

**G-2.2.5 Thymol Blue Indicator Solution**

## G-2.3 Procedure

Pipette 25 ml of styrene sample into a 250 ml glass-stoppered erlenmeyer flask containing 25 ml of methanol. Add 0.2 ml of thymol blue indicator and, if necessary, neutralize with 0.05 N sodium hydroxide or hydrochloric acid to the red-yellow end-point. Add 25 ml of the neutralized hydroxylamine hydrochloride solution and allow to stand for 1 h, shaking the flask occasionally. Titrate to the original red-yellow end-point with standard 0.05 N sodium hydroxide dispensed from a 10 ml burette, and record this value. Let it stand for another hour and again titrate any acid which may have been liberated.

## G-2.4 Calculation

Aldehyde content (as benzaldehyde), percent by

$$\text{mass} = \frac{V \times N \times 10.61}{25 \times 0.9}$$

where

$V$  = total volume of sodium hydroxide required for both titrations, in ml; and

$N$  = normality of standard sodium hydroxide solution used.

## G-3 METHOD B

### G-3.1 Outline of the Method

In this method 2,4-dinitrophenylhydrazine (DNPH) reacts with carbonyl groups (aldehydes) to form the corresponding hydrazone. The hydrazone has a yellow coloration in solution. The intensity of the yellow coloration is measured with a UV-spectrophotometer and the resulting absorbance is compared to a calibration curve for quantitation.

### G-3.2 Apparatus

**G-3.2.1 UV- Spectrophotometer**

**G-3.2.2 Sample Tube** — 24 mm

**G-3.2.3 Pipette**

**G-3.2.4 Microcentrifuge Tube**

**G-3.2.5 Stopwatch**

### G-3.3 Reagents

**G-3.3.1 Benzaldehyde**

**G-3.3.2 Acetonitrile** — HPLC grade

**G-3.3.3 Toluene** — HPLC grade

**G-3.3.4 2,4-Dinitrophenylhydrazine** — 30 percent water (H<sub>2</sub>O)

**G-3.3.5 Hydrochloric Acid (HCl)** — 6.0 N

**G-3.3.6 Benzaldehyde Stock Solution**

**G-3.3.7 2,4-Dinitrophenylhydrazine (DNPH) Stock Solution**

### G-3.4 Sample Preparation

**G-3.4.1** Pipette 60 ml of 2,4-dinitrophenylhydrazine (DNPH) into a volumetric flask. To it add 450 µl of 6 N hydrochloric acid using microcentrifuge tube, cap it and allow it to stand for 1 min to 2 min before use.

**G-3.4.2** Now pipette 15 ml of the prepared 2,4-dinitrophenylhydrazine solution into two clean and dry spectrophotometric cuvettes (one tube for blank and another one for the sample). Add 2 ml toluene to one cuvette (that is blank solution), and 2 ml of sample to the other cuvette (that is sample solution). Cap and mix both the solutions, start the stopwatch and wait exactly 5 min.

**G-3.4.3** Set the instrument as per instrument manufacturer's instructions. Place the blank cuvette into uv-spectrophotometer at 450 nm and press zero. Immediately, place the sample cuvette into the uv-spectrophotometer, then read and record the aldehyde concentration at 450 nm.



## ANNEX H

[Table 1, Sl No. (vii)]

## DETERMINATION OF PEROXIDES

## H-1 GENERAL

Three methods namely, Method A determination of peroxides by titrating material with titanous chloride solution, Method B determination of peroxides by titrating material with sodium thiosulfate solution and Method C determination of peroxides by UV-spectrophotometry have been prescribed. In case of disputes, Method C shall be the referee method.

## H-2 METHOD A

## H-2.1 Outline of the Method

A methanol solution of ferrous ammonium sulphate and potassium thiocyanate is acidified with dilute sulphuric acid and any ferric iron present is reduced by the careful addition of titanous chloride. A sample of styrene is then added and any peroxides present will oxidize the ferrous iron to ferric iron forming the red ferric thiocyanate colour. The ferric iron is titrated with titanous chloride that has been standardized freshly against ferric iron, and calculated as hydrogen peroxide. Any peroxide oxygen will be included.

## H-2.2 Apparatus

**H-2.2.1 Burette** — 50 ml long-tipped burette and, if desired, as assembly arranged for storing and using the titanous chloride solution under an inert atmosphere

## H-2.3 Reagents

## H-2.3.1 Titanous Chloride Solution

Mix 10 ml of concentrated hydrochloric acid and 6.5 ml of 20 percent titanous chloride and heat on steam bath in an inert atmosphere for 10 min. Dilute to 500 ml with distilled water. This solution should be standardized once a day and preserved under carbon dioxide atmosphere.

## H-2.3.2 Standard Iron Solution

Dissolve 35.11 g of crystallized ferrous ammonium sulphate  $[\text{FeSO}_4 \cdot (\text{NH}_4)_2 \cdot 6\text{H}_2\text{O}]$  in 400 ml of distilled water. Add 25 ml of concentrated sulphuric acid, warm the solution, and add potassium permanganate solution until the iron is completely oxidized. Dilute to 1 litre with distilled water. One

milliliter contains 0.005 g of ferric-iron.

## H-2.3.3 Ferrous Ammonium Sulphate

Dissolve 39.2 g of ferrous ammonium sulphate in 800 ml of distilled water. Add 50 ml of concentrated sulphuric acid and dilute to 1 litre with distilled water.

**H-2.3.4 Potassium Thiocyanate** — 10 percent aqueous solution

**H-2.3.5 Sulphuric Acid** — 1 : 1 aqueous solution

**H-2.3.6 Hydrochloric Acid** (see IS 265)

**H-2.3.7 Methanol** — absolute

**H-2.3.8 Standard Titanous Chloride Solution** — 0.2 N

Mix 150 ml of 20 percent titanous chloride solution with 100 ml of concentrated hydrochloric acid. Dilute to 1 litre with distilled water, Mix the solution thoroughly by passing a current of oxygen-free inert gas through it. If necessary, filter the solution through a filter paper until the filtrate is completely clear. Agitate the solution thoroughly with the inert gas for a few minutes and store the solution in the amber storage bottle with inert atmosphere. Standardize with ferric alum solution.

NOTE — For the preparation of 0.02 N standard solution from 20 percent titanous chloride solution, mix 15 ml of the 20 percent titanous chloride solution with 10 ml of 38 percent hydrochloric acid and proceed as above.

## H-2.4 Standardization of Titanous Chloride Solution

Pipette 10 ml of the standard iron solution into a 250 ml wide-mouth conical flask containing 50 ml of water, 15 ml of concentrated hydrochloric acid and 10 ml of 10 percent potassium thiocyanate. Add a small lump of solid carbon dioxide to the flask to maintain an inert atmosphere over the solution at all times. Keep the tip of the burette low in the flask and titrate with the titanous chloride solution until the pink colour of the ferric thiocyanate just disappears.

## H-2.4.1 Calculation

Hydrogen peroxide factor of titanous chloride, that is, mass in g of hydrogen peroxide equivalent to

1 ml of the titanous chloride solution, is calculated as:

$$\text{Factor} = (0.05 \times 17.01) / (T_8 \times 55.85)$$

where

$T_8$  = volume of titanous chloride required for the titration of 0.05 g of ferric iron contained in 10 ml of the standard iron solution, ml.

### H-2.5 Procedure

Pipette 10 ml of approximately 0.1 N ferrous ammonium sulphate into a 250 ml wide-mouth conical flask containing 100 ml of methanol, 5 ml of 10 percent potassium thiocyanate, and 2 ml of 1 : 1 sulphuric acid. Add a small lump of solid carbon dioxide to the flask, mix by swirling, cover with a watch-glass and allow to stand for 15 min. Carefully add the titanous chloride solution, while keeping the tip of the burette low in the flask, until the pink colour just disappears. This amount of titanous chloride used is not required in the calculation. Pipette 25 ml of the sample of styrene into the above adjusted solution, mix by swirling, cover with a watch-glass, and allow to stand for 2 h. Titrate with the standard titanous chloride solution until the pink color of the ferric thiocyanate just disappears.

### H-2.6 Calculation

Hydrogen peroxide, percent by mass =

$$\frac{T \times \text{Factor} \times 100}{25 \times 0.9}$$

where

$T$  = volume of standard titanous chloride required for the ferric iron produced by the peroxides in the sample, in ml; and

$\text{Factor}$  = mass of hydrogen peroxide equivalent to 1 ml of the titanous chloride, g (H-2.4.1).

## H-3 METHOD B

### H-3.1 Apparatus

**H-3.1.1 Erlenmeyer Flasks** — glass-stoppered, 500 ml, equipped with 300 mm Liebig condensers having inner and outer standard taper joints.

**H-3.1.2 Electric Hot Plate** — with totally enclosed heating unit

**H-3.1.3 Boiling Chips**

### H-3.2 Reagents

**H-3.2.1 Glacial Acetic Acid**

**H-3.2.2 Isopropyl Alcohol**

**H-3.2.3 Sodium Iodide-Isopropyl Alcohol Solution**

A saturated solution of sodium iodide in isopropanol (approximately 200 g NaI/l) is prepared.

**H-3.2.4 Sodium Thiosulfate (Standard Solution)** — 0.01 N

Dissolve 2.5 g of sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) and 0.1 g of sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) in water and dilute it to 1 litre. Standardize against primary standard potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ).

### H-3.3 Procedure

Take 200 ml of isopropyl alcohol into each of two 500 ml Erlenmeyer flasks containing several boiling chips. Add 10 ml of glacial acetic acid to each flask. Into one flask pipet 50 ml of the styrene monomer sample. One flask is to be labelled as 'sample' and the other flask as 'blank'. The condenser is fitted in place and heat the contents of both the flasks till solution starts boiling. Now pipette 50 ml of the saturated sodium iodide isopropyl alcohol solution into each flask and continue boiling gently for 10 min.

After this time, the flasks are removed from the heat source. Rinse each condenser with two 10 ml water by adding the rinsing to the respective flasks. Cool the flasks to room temperature. Titrate the liberated iodine in each flask with 0.01 N sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) solution to a light yellow color and continue to titrate slowly until the yellow color just disappears.

### H-3.4 Calculation

Calculate the peroxide content of the specimens as hydrogen peroxide, in parts per million (mg/kg) as follows:

$$\text{Peroxides, ppm (mg/kg)} = \frac{(A-B) \times N \times 1.7 \times 10000}{50 \times C}$$

where

$A$  = total volume of sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) solution required for titration of the specimen, in ml;

$B$  = total volume of sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) solution required for titration of the blank, in ml;

$N$  = normality of sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) solution used; and

$C$  = density of styrene monomer at temperature pipetted (an approximate density of 0.9 may be used to determine the sample weight).

## H-4 METHOD C

### H-4.1 Outline of the Method

A yellow colouration is developed upon addition of styrene containing peroxides to tetrabutylammonium iodide (TBAI). The intensity of the yellow colouration is measured with a UV-spectrophotometer and quantified by the UV-spectrophotometer by using the slope factor derived from the calibration curve.

### H-4.2 Apparatus

#### H-4.2.1 UV-Spectrophotometer

#### H-4.2.2 Sample Tube — 24 mm

#### H-4.2.3 Vortex Mixer

#### H-4.2.4 Pipettes

#### H-4.2.5 Stopwatch

### H-4.3 Reagents

#### H-4.3.1 Benzoyl Peroxide

#### H-4.3.2 *N,N*-Dimethylformamide (DMF)

#### H-4.3.3 Tetrabutylammonium Iodide (TBAI)

#### H-4.3.4 Toluene — HPLC grade

#### H-4.3.5 Glacial Acetic Acid

#### H-4.3.6 Ethyl Acetate — HPLC grade

#### H-4.3.7 Peroxide Stock Solution

### H-4.4 Procedure

**H-4.4.1** Prepare a blank solution by pipetting 5.0 ml ethyl acetate, 1 250  $\mu\text{l}$  glacial acetic acid and 5.0 ml of toluene into a volumetric flask and mix thoroughly. Now prepare the sample solution by pipetting 5.0 ml ethyl acetate, 1 250  $\mu\text{l}$  glacial acetic acid and 5.0 ml of sample into another volumetric flask and mix thoroughly. Add 1 250  $\mu\text{l}$  tetrabutylammonium iodide (TBAI) to blank and sample solution, cap it and mix the contents for approximately 5 min.

**H-4.4.2** Set the instrument as per instrument manufacturer's instructions. Place the prepared blank solution into uv-spectrophotometer at 450 nm and press 'CAL' (auto zero). Now place the sample into the uv-spectrophotometer at 450 nm. Wait for the reading to stabilize and record.

## ANNEX J

[Table 1, *Sl No.* (viii)]

### DETERMINATION OF SULPHUR

### J-1 GENERAL

Three methods namely, Method A Combustion method; Method B Reduction method and IS 1448 (Part 160) have been prescribed for determination of sulphur. In case of disputes, IS 1448 (Part 160) shall be the referee method.

### J-2 METHOD A

#### J-2.1 Outline of the Method

A known quantity of the material is burnt in a lamp. The products of combustion being drawn through sodium peroxide solution and the resulting sulphate being determined by nephelometric method.

### J-2.2 Apparatus

#### J-2.2.1 Air purifying train

It shall consist of two bottles *A* and *B* and a tower *C* of the dimensions shown in Fig. 3, all packed with pumice. The pumice in *A* and *C* shall be soaked with potassium hydroxide solution, so that its depth in the former is 2 cm to 3 cm, and in the latter just sufficient to cover the inlet tube. The pumice in *B* shall be soaked with lead acetate solution to a depth of 2 cm to 3 cm.

The inlet tubes in *A* and *B* shall reach within a few millimeters of the bottom.

This arrangement removes acidic sulphur compounds only and if the air contains such compounds as carbon disulphide or thiophene, the train shall be preceded by a length of combustion tubing, packed, with coarse copper oxide, and heated to dull redness. In that case, the bottle *B* shall contain potassium hydroxide solution instead of lead acetate solution.

#### J-2.2.2 Chimney and absorption train

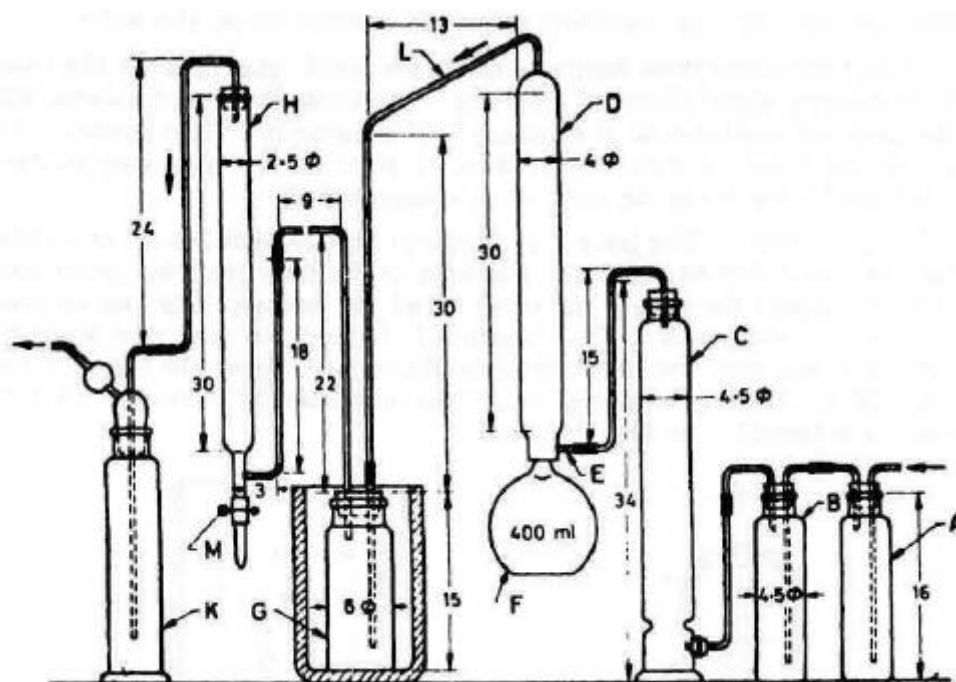
The air from the tower *C* shall be introduced by the side-tube *E* to the foot of the chimney *D*, into which the lamp *F* fits with a rubber stopper. The products of combustion shall be drawn up the chimney *D* and from there into the first absorption bottle *G*, which shall consist of a 500 ml wide-mouthed bottle, immersed in an ice-bath. The connection shall be sloped at *L* to avoid the possibility of condensed water running down the hot chimney. The ice-bath condenses the water and also cools the gases before they enter the sodium peroxide solution, thus

avoiding undue decomposition of the latter.

From the absorption bottle *G*, the gases shall pass through the tower *H*, containing short pieces of glass rod. The train shall be completed with the drechsel wash-bottle *K* of about 250 ml capacity. The bottles *G* and *K*, and the tower *H* shall each contain 25 ml of sodium peroxide solution. The liquid from *H* can be drained by pinch-cock *M*.

#### J-2.2.3 Lamp,

*F* as shown in Fig. 4A shall consist of a round flask of about 400 ml capacity. The neck of the flask shall be constricted at *N* to support the tube *R* in which shall be loosely fitted an ordinary cylindrical cotton-wick. The length of *F* shall be such that when the lamp is in position, the top *R* shall be about 4 cm above the top of *E* (see Fig. 4B). During weighing, etc, a test tube *P* shall be inverted over the wick as a sheath (see Fig. 4A).



All dimensions in centimeters  
FIG. 3 AIR PURIFYING TRAIN

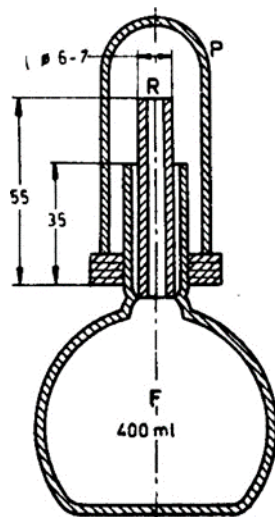


FIG. 4A

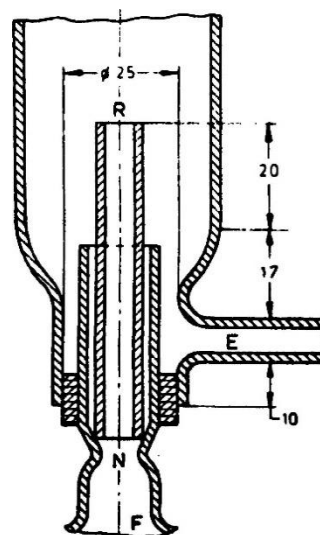


FIG. 4B

All dimensions in millimeters  
FIG. 4 LAMP

**J-2.2.4 Nessler tubes**, two tubes of thin transparent glass, about 25 mm in diameter and about 150 mm in length, graduated up to 50 ml. The depth, measured internally from the graduated mark to the bottom, shall not vary by more than 2 mm in the tubes used.

### J-2.3 Regents

**J-2.3.1 Absolute Alcohol** (see IS 321)

**J-2.3.2 Potassium Hydroxide Solution** — 50 percent (m/v)

**J-2.3.3 Lead Acetate Solution** — 10 percent (m/v)

**J-2.3.4 Sodium Peroxide Solution**

Dissolve cautiously 10 g of sodium peroxide in 500 ml of ice-cold water

**J-2.3.5 Concentrated Hydrochloric Acid** — specific gravity 1.16 (see to IS 265)

**J-2.3.6 Barium Chloride Solution** — 12 percent (m/v) of barium chloride crystals dissolved in water

**J-2.3.7 Standard sulphate solution**

Acidify 75 ml of sodium peroxide solution with concentrated hydrochloric acid, bring to boil and then cool. Transfer the solution to a 500 ml volumetric flask. Add 15.6 ml of 0.01 N standard

sulphuric acid and make up the volume to 500 ml mark

### J-2.4 Procedure

#### J-2.4.1 Cleaning of Wick

Boil the wick for an hour with soap solution to remove oily matter and size (which might contain sulphur compounds), then boil it for several minutes with successive changes of distilled water until the water remains clear and bright. Squeeze out the wick, wash it twice in absolute alcohol, dry at  $(100 \pm 2)^\circ\text{C}$  and insert in tube R.

**J-2.4.2** Transfer 250 g of the material to the lamp F, pouring a little down the tube R, replace the sheath and weigh. Attach the wash-bottle K to a suction pump, light the lamp, draw a rapid stream of air through the apparatus and quickly push the lamp into position. The initial stream of air shall not be too slow as otherwise the lamp may be extinguished. Once the test is under way, reduce the air stream a little but take care that it is rapid enough to keep the lamp burning vigorously. When all the sample has been burned, which will take about 24 h, stop the flow of air through the apparatus, remove the lamp, replace the sheath and weigh. The difference between the two weighing, M, is the mass of material consumed.

**J-2.4.3** Transfer contents of the vessels G, H and K to a beaker. If necessary, boil until the volume of the

liquid is reduced to about 450 ml, then add 10 ml of concentrated hydrochloric acid. Filter and again bring it to boil. Cool the solution and make up the volume to 500 ml in a volumetric flask.

**J-2.4.4** Pipette out 50 ml of the test solution (**J-2.4.3**) into a nessler tube and add 2 ml of barium chloride solution. Carry out a control test in another nessler tube using 50 ml of standard sulphate solution if *M* is equal to 250 g (or a proportionate lower or higher volume) and 2 ml of barium chloride solution. Mix both the solutions well, stir and compare the turbidity produced, if any, after 5 min.

The material shall be considered to have satisfied the specified requirement if the turbidity produced in the test with the material is not greater than that produced in the control test.

### J-3 METHOD B

#### J-3.1 Outline of the Method

The sulphur present in the material is reduced with raney nickel to metal sulphide, which is heated with hydrochloric acid. The hydrogen sulphide evolved is absorbed in sodium hydroxide solution and titrated with standardized mercuric acetate solution in the presence of dithizone as indicator. This method determines all sulphur compounds normally encountered in methyl alcohol for industrial uses. It does not, however, determine alkyl sulphur and other oxygen bounded sulphur compounds.

#### J-3.2 Apparatus

**J-3.2.1** *Reduction Apparatus* (see Fig. 5)

**J-3.2.2** *Burette* — class A, 10 ml capacity (see IS 1997) and graduated in 0.05 ml

#### J-3.3 Reagents

**J-3.3.1** *Nitrogen gas* — free from carbon dioxide

**J-3.3.2** *Acetone* (see IS 170)

**J-3.3.3** *Raney Nickel* — treat 10 g of 50/50 nickel/aluminium alloy with 100 ml of 10 percent (*m/v*) sodium hydroxide solution, cooling with ice while the reaction proceeds. Wash the raney nickel three times with water, remove the water by decantation and store the Raney nickel under 100 ml of isopropyl alcohol. Raney nickel prepared in this way retains its activity for about three days.

Five millilitres of this suspension will reduce quantitatively 1.0 mg to 1.5 mg of organic sulphur to hydrogen sulphide.

A stabilized form of raney nickel is available commercially and may be prepared by warming half pellet (0.24 g) in the reaction flask, in 10 ml of acetone. When the pellet has disintegrated, decant the acetone and introduce the sample.

NOTE — Raney nickel is spontaneously flammable in air.

**J-3.3.4** *Sodium Hydroxide Solution* — 1.0 N

**J-3.3.5** *Hydrochloric Acid* — (specific gravity 1.18) diluted (3 : 2) with water

**J-3.3.6** *Mercuric Acetate Solution*

Dissolve 0.200 g of mercuric oxide in 25 ml of water containing 1 ml glacial acetic acid and dilute to 1 000 ml with water and standardize as follows:

**J-3.3.6.1** *Standardization of the Mercuric Acetate Solution*

Add a known amount of an aliphatic sulphur compound (di-isoamyl sulphide) to the sulphur-free methanol. Carry out a determination by the method (**J-3.4**) using an aliquot of this solution representing 130 µg to 170 µg of sulphur. From the volume of the mercuric acetate solution required in the titration, calculate the mass, in micrograms, of sulphur equivalent to 1 ml of the mercuric acetate solution.

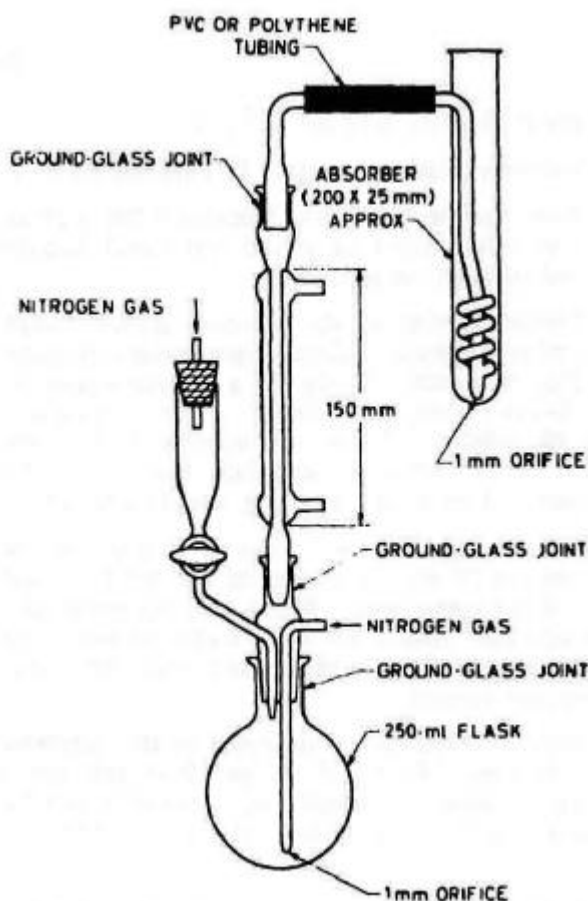


FIG. 5 REDUCTION APPARATUS FOR USE IN THE DETERMINATION OF SULPHUR AND SULPHUR COMPOUNDS

**J-3.3.7 Methanol, Sulphur-Free** — into a 2 litres, two-necked round bottom flask, measure 20 ml of acetone and add 0.5 g (1 pellet) of Raney nickel or 10 ml of the suspension. Warm until the pellet has disintegrated and decant the acetone. Add 1 500 ml of methanol and reflux on a water-bath. Insert the nitrogen lead in the second neck and pass in gas at the rate of 2 bubbles per second. Heat until slight effervescence is observed on the methanol surface and continue for 30 min. Turn off the gas flow and the heating and allow the nickel to settle. Decant the methanol into a flask and distil. Reject the first 250 ml and collect the next 1 000 ml, rejecting the remainder.

**J-3.3.8 Dithizone** — one percent (*m/v*) solution in acetone

#### J-3.4 Procedure

Place 5 ml of raney nickel suspension, or half pellet as described in the flask, then add 100 ml of the sample. Place the flask on a water-bath and pass

nitrogen through at the rate of about 3 l/h. Adjust the temperature of the heating mantle until slight effervescence appears on the surface (just below boiling point). Continue for 50 min, swirling the flask at intervals.

Place 10 ml of the sodium hydroxide solution and 10 ml of acetone in the absorber.

Place 15 ml of the hydrochloric acid in the dropping funnel, switch off the heating mantle, and attach a second nitrogen lead to the glass tube in the top of the dropping funnel. Admit the acid, a few milliliters at a time, to the flask. Add two drops of the dithizone solution and one drop of the mercuric acetate solution to the absorber using the burette. The colour change should be from yellow to pink. Liberation of hydrogen sulphide reverses the colour change, when all the acid has been added, increase the nitrogen flow rate to about 12 litres per hour, switch on the heating mantle again, continue heating for a further 20 min, titrating with the mercuric acetate solution as necessary. When all the hydrogen sulphide has

apparently been titrated, disconnect the flow of nitrogen momentarily, cool the flask by blowing air on it and allow the absorbent to travel up the delivery tube in the absorber, to recover any hydrogen sulphide which may be absorbed on its surface.

Carry out a blank test on the raney nickel and the sulphur-free methanol.

### J-3.5 Calculation

Sulphur and sulphur compounds are calculated as:

$$S, \text{ parts per million (ppm) by mass} = \frac{M \times (V_2 - V_1)}{100 \times S}$$

where

$M$  = mass of sulphur equivalent to 1 ml of the mercuric acetate solution, in  $\mu\text{g}$  (J-3.3.6.1);

$V_2$  = volume of the mercuric acetate solution required in the test determination, in ml;

$V_3$  = volume of the mercuric acetate solution required in the blank test, in ml; and

$S$  = specific gravity of the sample.

## ANNEX K

[Table 1, Sl No. (ix)]

### DETERMINATION OF CHLORIDES

#### K-1 GENERAL

Two methods namely, Method A determination of chloride by turbidimeter and Method B determination of chloride by microcoulometer have been prescribed. In case of disputes, method B shall be the referee method.

#### K-2 METHOD A

##### K-2.1 Outline of the Method

A sample of the material is mixed with alcohol to aid combustion and is burnt in chloride free air. The products of combustion are bubbled through sodium hydroxide solution which, retains the chlorides as sodium chloride. The acidity and the volume of the absorbing solution are adjusted, the chloride precipitated as silver chloride, and the turbidity of the solution determined by means of a suitable turbidimeter.

##### K-2.2 Apparatus

###### K-2.2.1 Air Purification

The air is passed first through a 2.5 cm  $\times$  30 cm straight drying tube filled with coarse calcium chloride, then through a 2.5 cm  $\times$  85 cm quartz or vycor glass tube packed with 50 cm of coarse cupric oxide (CuO), 15 cm of asbestos impregnated with lead chromate, and a 15 cm roll of copper gauze. The cupric oxide portion of the tube is heated to approximately 700 °C in an electrical resistance furnace. The air is finally passed through a large manifold reservoir, the size of which depends on the number of lamps used. The larger the reservoir, the

more uniform is the flow of air to all lamps and fewer burner manipulations are required.

**K-2.2.2 Lamp** — consisting of a burner, chimney, absorber, and condenser and the inner tube of the lamp uses 5 full strands of the cotton

**K-2.2.3 Alcohol Lamp** — for igniting the burner

**K-2.2.4 Suitable Turbidimeter** — used for water analysis or any other turbidimeter may also be used which shall be accurate enough as to give readings

#### K-2.3 Reagents

##### K-2.3.1 Sodium Hydroxide Absorbing Solution

Dissolve 10 g of sodium hydroxide in 1 litre of distilled water

##### K-2.3.2 Nitric Acid — 1 N

##### K-2.3.3 Phenolphthalein Indicator (see IS 2263)

##### K-2.3.4 Ethanol (see IS 321)

##### K-2.3.5 Nitric acid — Silver Nitrate Reagent

Prepare a solution approximately 0.2 N of nitric acid and 0.01 N of silver nitrate by diluting 12.5 ml of concentrated nitric acid to 1 litre and adding 1.70 g of silver nitrate (see IS 2214)

##### K-2.3.6 Standard Chloride Solution

Dilute to 1 litre with distilled water 10 ml of a solution containing 4.12 g of reagent grade sodium chloride per litre. This solution contains 25 mg of chloride per litre. 1 ml diluted to 50 ml, as in the



procedure, gives a solution corresponding to 0.5 ppm of chloride.

#### K-2.4 Procedure

Place 30 ml of the sodium hydroxide absorbing solution in the absorber. Adjust the cotton lamp wick until it is slightly above the rim of the burner, and with the burner inserted in the chimney, adjust the flow of air to the approximate velocity necessary for burning. Remove the burner and add about 25 ml of a mixture consisting of 15 ml of styrene sample in 40 ml of absolute alcohol. Weigh the lamp plus all the sample. Ignite the wick with the flame of an alcohol lamp and re-insert the burner in the chimney. Adjust the air flow to give a clear and smokeless flame.

When about 20 ml of the mixture has been burnt, reweigh the lamp and the residual unburnt portion of the sample, and transfer the absorbing solution to a 400 ml beaker. Rinse the apparatus several times with distilled water. The final volume should be about 200 ml. Evaporate to about 10 ml on a hot plate in a fume hood free from chloride fumes. Filter the evaporated solution through a filter paper into a 50 ml volumetric flask, washing the beaker several times with distilled water. Make the filtrate neutral to phenolphthalein with 1 N nitric acid. The resulting volume should approximate 200 ml. Add 20 ml of ethanol. The bicarbonate present may cause the solution to become turbid at this point, if, so, carefully add 1 N nitric acid dropwise until all of this turbidity is dispelled. Add dropwise 5 ml of the nitric acid-silver nitrate reagent, then make up to the mark with ethanol and shake. Place the flask in a water-bath at 40 °C for 35 min, then cool the sample rapidly to room temperature.

Transfer the contents of the flask to a 20 mm optical cell of the turbidimeter. Stir in an additional 10 ml of ethanol and determine the turbidity. Compare the readings against the standard curve to find parts per million of chloride. Concentrations of greater than 5 ppm chloride should be determined on aliquots of the sample. Determine a blank test on the apparatus and air stream, following the same procedure described above but omitting the sample. Burn the blank for the average length of the time required for the sample.

Prepare the standard chloride calibration curve by measuring out aliquot portions of the standard chloride solution and analyzing by the above procedure.

#### K-2.5 Calculation

$$\text{Chloride (as Cl), percent by mass} = \frac{(A-B) \times 0.1}{S}$$

where

- A = chloride of sample, in mg;
- B = chloride of blank, in mg; and
- S = sample burnt, in g.

### K-3 METHOD B

#### K-3.1 Outline of the Method

A sample is injected into a combustion tube maintained at 900 °C, having a flow of oxygen and argon carrier gas. Oxidative pyrolysis converts the organic halides to hydrogen halides that then flow into a titration cell where it reacts with silver ions present in the electrolyte. The silver ion thus consumed is coulometrically replaced and the total electrical work to replace it is a measure of the organic halides in the specimen injected.

#### K-3.2 Apparatus

##### K-3.2.1 Microcoulometer

##### K-3.2.2 Syringe — 50 µl capacity

#### K-3.3 Reagents

##### K-3.3.1 Chloride Standard Stock Solution

Weigh accurately 0.093 g ( $\pm 0.0001$  g) of 2,4,6-trichlorophenol. Transfer it to a 500 ml volumetric flask and dilute it up to the mark with methanol

**K-3.3.2 Solvent** — any suitable solvent capable of dissolving the chloride sample (for example, methanol, isooctane, toluene, *p*-xylene)

#### K-3.4 Calibration

**K-3.4.1** Set the instrument as per manufacturer's instruction. Now, pipette 1 ml, 15 ml and 30 ml of chloride stock solution into 100 ml volumetric flask and dilute it up to the mark with solvent. The standards prepared are of approximately 1 µg Cl/ml, 15 µg Cl/ml and 30 µg Cl/ml.

NOTE — Sample size for both standard solution and sample to be injected is 80 percent or less of the syringe capacity.

**K-3.4.2** Using syringe, inject the above prepared standard in the instrument and prepare a three-point curve using the instrument manufacturer's recommendations.

### K-3.5 Procedure

Clean the syringe to be used for the sample. Flush it several times with the sample. Now take the sample in the syringe and inject the sample

### K-3.6 Calculation

Calculate results utilizing volume and known specific gravity in milligrams per kilograms as

follows:

$$\text{Chloride, mg/kg} = \frac{M-B}{V \times D} \times \frac{1}{\text{recovery factor}}$$

where

- $M$  = measured chloride value, in  $\mu\text{g}$ ;  
 $B$  = blank chloride value, in  $\mu\text{g}$ ;  
 $V$  = sample injection volume, in ml;  
 and  
 $D$  = density of the sample

$$\text{Recovery factor} = \frac{\text{chlorides titrated, g}}{\text{theoretical value, g}}$$

## ANNEX L

[Table 1, SI No. (x)]

### DETERMINATION OF POLYMER CONTENT

#### L-1 GENERAL

Three methods namely, Method A determination of polymer content by turbidimeter or colorimeter, Method B determination of polymer content by spectrophotometer or photometer and Method C determination of polymer content by visual method have been prescribed. In case of disputes, method B shall be the referee method.

#### L-2 METHOD A

##### L-2.1 Outline of the Method

This method is intended to determine trace quantities of polymer in styrene. When the styrene is added to dry methanol, the polymer precipitates and the resultant turbidity is quantitatively estimated by means of a turbidimeter or calorimeter.

##### L-2.2 Apparatus

###### L-2.2.1 Turbidimeter or Colorimeter

###### L-2.2.2 Pipette — 25 ml and 10 ml

##### L-2.3 Reagents

###### L-2.3.1 Methanol

###### L-2.3.2 Standard Polymer Solution

Fill a 20 mm  $\times$  175 mm test tube one third full of inhibited styrene and place it in an oven at 150 °C for about 60 h. After the styrene has polymerized, allow the polymer to age at room temperature for three or four days. Dissolve 0.450 0 g of the styrene polymer in freshly distilled polymer-free inhibited

styrene in a 100 ml volumetric flask. When the polymer is dissolved, dilute to volume with additional polymer-free styrene. This solution contains 5 000 ppm ( $m/m$ ) polymer.

Dilute further to yield solutions containing 5 ppm ( $m/m$ ), 10 ppm ( $m/m$ ), 15 ppm ( $m/m$ ), 20 ppm ( $m/m$ ), ....50 ppm ( $m/m$ ) of polymer.

#### NOTES

1 Standard polymer solution may also be prepared by any other alternate methods.

2 Toluene may also be used as an alternate to polymer-free inhibited styrene while preparing standard polymer solution.

##### L-2.4 Procedure

Take 20 ml of dry methanol in a 100 ml glass stoppered flask, add 20 ml of standard polymer solution and shake vigorously for 30 s and allow it to stand for 5 min. Read the percent transmission on the colorimeter or turbidimeter using dry methanol and polymer free styrene as blank at 470 nm. Draw a graph of percent transmission against ppm polymer. To estimate polymer in the given sample of styrene, follow the above procedure and read off polymer, ppm against percent transmission observed, from the standard graph prepared.

#### NOTES

1 If the precipitate is in excess, dilute the original sample to a suitable range with pure styrene, toluene or benzene and follow the above procedure. Then apply the dilution factor to the result to compute the polymer content in the original sample.

**L-3 METHOD B****L-3.1 Outline of the Method**

This method is intended to determine polymer content in styrene by measuring the turbidity produced when dry methanol is added to styrene sample.

**L-3.2 Apparatus**

**L-3.2.1 Spectrophotometer or photometer** — with cells having 50 mm to 150 mm light path

**L-3.2.2 Pipette** — 10 ml, 15 ml

**L-3.2.3 Bottle or Flasks** — with glass stoppers

**L-3.3 Reagents**

**L-3.3.1 Hexane** — dry

**L-3.3.2 Methanol** — dry

**L-3.3.3 Toluene** — dry

**L-3.3.4 Polystyrene**

Take 50 ml styrene monomer and washed it twice with 50 ml of 1 N sodium hydroxide and twice with 50 ml water, using separating funnel. Now filter the styrene obtained after second water wash through two layers of rapid filtering folded filter paper. To promote polymerization, take 20 ml of this styrene monomer into the test tube and heat in an oven at 100 °C for 24 h. At the end remove the polystyrene from the test tube and grind it to fine powder.

NOTE — Commercially available high purity polystyrene may also be used.

**L-3.3.5 Styrene Polymer Standard Solution**

**L-3.3.5.1** Weigh 0.090 5 g of polystyrene (**L-3.3.4**) and dissolve it in 1 000 ml of toluene measured at 25 °C. The solution contains 100 mg/kg of polymer in styrene. The solution as prepared is the standard for polymer in styrene.

**L-3.3.5.2** Take 1 ml, 3 ml, 6 ml, 9 ml, 12 ml and 15 ml of 100 mg/kg styrene polymer standard (**L-3.3.5.1**) solution in volumetric flasks and dilute them with 100 ml toluene. The solution as prepared contains 1 mg/kg, 3 mg/kg, 6 mg/kg, 9 mg/kg, 12 mg/kg and 15 mg/kg of styrene polymer.

**L-3.4 Calibration**

**L-3.4.1** Pipette 15 ml of dry methanol and 10 ml of each polymer standard solution (**L-3.3.5.2**) into a series of bottle or flask, with glass stopper and mix thoroughly. Now in another series of bottle or flask, with glass stopper, pipette 15 ml of hexane and

10 ml of each polymer standard solution (**L-3.3.5.2**) and mix thoroughly.

**L-3.4.2** Allow all the solutions prepared above to stand for  $(15 \pm 1)$  min. After this time, pour the solutions into spectrophotometer cells and measure the absorbance of each solution at 420 nm, using the series of standard hexane mixtures as blank.

**L-3.4.3** Plot the calibration curve by plotting the absorbance versus milligrams per kilogram of polymer.

**L-3.5 Procedure**

**L-3.5.1** Set the instrument as per instrument manufacturer's instructions. Pipette 15 ml of hexane into one bottle or flask and in another bottle or flask, pipette 15 ml of methanol. Add 10 ml sample in both bottles or flask and mix thoroughly.

**L-3.5.2** To measure absorbance, proceed by following (**L-3.4.2**) by using sample hexane mixture as blank.

**L-3.5.3** Calculate the milligram per kilogram of polymer from calibration curve.

**L-4 METHOD C****L-4.1 Outline of the Method**

This test method determines polymer content by visual observing degree of turbidity produced on addition of methanol to sample.

**L-4.2 Apparatus**

**L-4.2.1 Test Tube** — 25 x 125 mm

**L-4.2.2 Pipette** — 2 ml and 10 ml

**L-4.2.3 Daylight Fluorescent Tube** — equipped with curved reflector

**L-4.3 Reagents**

**L-4.3.1 Methanol** — dry

**L-4.3.2 Polystyrene** — uncoloured, unlubricated (see **L-3.3.4**)

**L-4.3.3 Toluene** — dry

**L-4.4 Procedure**

**L-4.4.1** Pipette 2 ml of sample into a clean, dry test tube. To it add 10 ml of dry methanol and stopper the test tube with a cork covered with aluminum foil and shake vigorously for a few seconds.

**L-4.4.2** After shaking test tube, visually inspect the mixture by looking through it toward a source of artificial daylight with 10 ppm polymer standard.

#### L-4.5 Report

Report less than 10 ppm if “faint trace of cloudiness

in comparison with pure dry methanol" or Nil if "no cloudiness observed by comparison with pure dry methanol".

## ANNEX M

[Table 1, Sl No. (xii)]

### DETERMINATION OF INHIBITOR CONTENT

#### M-1 GENERAL

Two method namely, Method A determination of inhibitor content by colorimeter and Method B determination of inhibitor content by spectrometer have been prescribed. In case of disputes, Method B shall be the referee method.

#### M-2 METHOD A

##### M-2.1 Outline of the Method

This method determines the amount of 4-tert-butylcatechol (TBC) (also known as 4-*t*-butyl-1, 2-dihydroxybenzene) added to styrene to prevent polymerization while in transit and storage. It determines only the active inhibitor. Catechols in alkaline solution readily absorb oxygen, with the formation of highly coloured compounds. TBC gives a pink to blood-red colouration, that is, proportional to the amount of catechol present. The colour is apparently caused by the oxidation of the dihydroxy molecule to the quinone. This is measured using nessler tube or any suitable calorimeter.

##### M-2.2 Apparatus

###### M-2.2.1 Colorimeter

###### M-2.2.2 Pipette — 50 ml

###### M-2.2.3 Separating Funnel — 125 ml

##### M-2.3 Reagents

###### M-2.3.1 Sodium Hydroxide Solution — 1 N

###### M-2.3.2 Standard Inhibitor Solution

In 1 000 ml of uninhibited styrene prepared by washing 4 volumes of styrene three times with one volume of 10 percent aqueous solution of sodium hydroxide, dissolve 0.113 g of TBC. This solution shall contain 125 ppm of TBC.

##### M-2.3.3 Styrene Uninhibited

Wash 4 volumes of styrene three times with one volume of 10 percent reagent grade sodium hydroxide. Refrigerate to prevent polymerization.

##### M-2.4 Procedure

Prepare standard solutions containing 0 ppm, 5 ppm, 10 ppm, 15 ppm, 100 ppm TBC by adding 0 ml, 2 ml, 4 ml, 6 ml . . . 40 ml of standard TBC solution, respectively to 125 ml separating funnels. Add sufficient inhibitor-free styrene to make a total volume of 50 ml. To each separating funnel, add 50 ml of 1.0 N caustic soda solution and shake for 30 s to 60 s and allow the two layers to separate. Repeat the shaking of the separating funnel three to five times and finally allow clear separation to take place. Filter the aqueous layer through a coarse filter paper into a 100 ml Nessler tube. Then measure the percent transmission on the colorimeter at 490 nm within 5 min, observing all the necessary precautions and instructions of the instrument, using 1.0 N caustic soda solution as blank. Draw a graph of percent transmission against TBC, ppm.

Take 50 ml of styrene in a separating funnel. Add 50 ml of 1.0 N caustic soda solution and shake for 30 s to 60 s. Allow the two layers to separate. Repeat the shaking of the separating funnel three to five times and finally allow clear separation to take place. Filter the aqueous layer through a coarse filter paper into a 100 ml glass tube. Then measure the percent transmission on the colorimeter at 490 nm within 5 min observing all the necessary precautions and instructions of the instrument using 1.0 N caustic soda solution as blank.

Compare the reading with the standard graph and read the amount of TBC in the sample.

NOTE — If the concentration of TBC goes out of the range

of the standard curve, take a smaller sample and find out the percent transmission observing the above procedure. The result must be corrected by the factor (F) to give the actual concentration of TBC:

$$\text{Factor (F)} = \frac{50}{\text{sample size (ml)}}$$

## M-3 METHOD B

### M-3.1 Apparatus

**M-3.1.1 Visible Range Spectrophotometer** — with absorption cell having light paths 1 cm to 5 cm

**M-3.1.2 Volumetric Pipettes**

### M-3.2 Reagents

**M-3.2.1 4-tert-butylcatechol**

**M-3.2.2 Toluene**

**M-3.2.3 Methanol**

**M-3.2.4 n-Octanol**

**M-3.2.5 Alcoholic Sodium Hydroxide**

Take 0.75 ml 10 N aqueous sodium hydroxide in volumetric flask and mix it with 25 ml methanol. With stirring, add 25 ml n-octanol and 0.75 ml of water. The reagent prepared is store in amber glass bottle. The reagent as prepared is stable for atleast 2 months

**M-3.2.6 4-tert-butylcatechol Stock Standard**

To prepare a stock solution, weigh  $(0.500 \pm 0.001)$  g TBC in  $500 \pm 1$  g of toluene in volumetric flask. The solution as prepared has concentration of approximately 1 000 mg/kg TBC in toluene. Exact. While exact concentration may be calculated as:

$$\text{mg TBC/kg Toluene} = \frac{\text{mass of TBC (in g)} \times \text{putiy of TBC} \times 10^6}{\text{mass of toluene (in g)}}$$

### M-3.3 Calibration

**M-3.3.1** Take 0.5 ml, 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, 7 ml and 100 ml of TBC and dilute them up to 100 ml with toluene. The standard as prepared above has concentration of approximately 5 mg/kg, 10 mg/kg, 20 mg/kg, 30 mg/kg, 40 mg/kg, 50 mg/kg, 70 mg/kg and 10 mg/kg TBC in toluene respectively.

**M-3.3.2** Determine the absorbance of one reagent blank and each standard solution as prepared at **M-3.3.1** by procedure as given at **M-3.4**.

**M-3.3.3** Plot the absorbance versus concentration. The plot as prepared shall be close to a straight line. If not, repeat the calibration with spectrometer cell that has less path length.

### M-3.4 Procedure

**M-3.4.1** Set the instrument as per instrument manufacturer's instructions. Zero the spectrometer with sample.

**M-3.4.2** Take 15 ml of sample in a clean flask. Pipette 300 µl of alcoholic sodium hydroxide in the above mentioned flask and mix the solution vigorously either by shaking or by using vortex mixer for 30 s. Now add 600 µl of methanol to the flask and shake for 15 s, creating clear solution from reaction emulsion. Measure the absorbance at 490 nm within 5 min. Measure the concentration in mg TBC/kg Toluene from the graph and calculate inhibitor content by using formula as given at **M-3.5** for density correction.

### M-3.5 Calculation

Inhibitor Content, mg /kg =

$$\frac{\text{Toluene density}}{\text{Sample density}} \times \text{Value measured from graph}$$

## ANNEX N

(Clause 5)

### SAMPLING OF STYRENE

#### N-1 GENERAL REQUIREMENTS OF SAMPLING

**N-1.1** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**N-1.2** Samples shall not be taken in an exposed place.

**N-1.3** The sampling instrument shall be clean and dry and shall be made of low or reduced spark generating material.

**N-1.4** The samples, material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

**N-1.5** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution.

**N-1.6** The samples shall be placed in suitable, clean, dry and air-tight glass containers preferably of amber or blue colour.

**N-1.7** The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

**N-1.8** Rubber stoppers, or composition cork shall not be used for closing the sample bottles.

**N-1.9** Sealing wax or other plastic sealing material, if used, shall be applied in such a way that it does not contaminate the sample when the bottles are opened.

**N-1.10** Each sample container shall be protected by covers of oil proof paper, metal foil, viscose or other suitable impervious material over the stopper to keep the moisture and dust away from the mouth of the bottle and to protect it while being handled.

**N-1.11** Each sample container shall be sealed airtight with a suitable stopper after filling and marked with full details of sampling, such as the grade of styrene, the date of sampling, the year of manufacture of the material, the batch number, the name of the sample, etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample.

## N-2 SAMPLING INSTRUMENT

The following forms of sampling instruments may be used:

- a) Sampling bottle or can for taking samples from various depths in large tanks; and
- b) Sampling tube.

### N-2.1 Sampling Bottle or Can

It consists of a weighed bottle or metal container with removable stopper or top, to which is attached a light chain (*see* Fig. 6). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

### N-2.2 Sampling Tube

It is made of metal or thick glass and is about 20 mm to 40 mm in diameter and 400 mm to 800 mm in length (*see* Fig. 7). The upper and lower ends are conical and reach 5 mm to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and is finally closed and withdrawn.

**N-2.2.1** For small containers, the size of the sampling tube may be altered suitably.

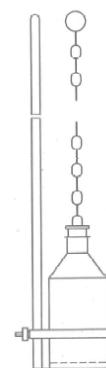
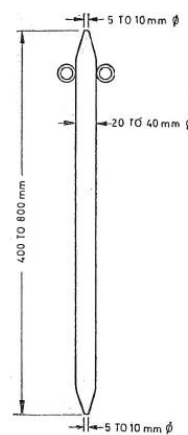


FIG. 6 SAMPLING BOTTLE OR CAN



All dimensions in millimeter

FIG. 7 SAMPLING TUBE

## N-3 SCALE OF SAMPLING

### N-3.1 Lot

In any consignment, all the containers of same size and grade drawn from the same batch of manufacture shall constitute a lot.

**N-3.2** Tests shall be conducted on each lot of each grade of styrene, separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col (2) and (3) of Table 5.

**N-3.3** The containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedure given in IS 4905 may be followed.

#### N-4 PREPARATION OF TEST SAMPLES

**N-4.1** From each of the containers selected according to **N-3.3**, a small representative's portion of the material not less than 600 ml, shall be drawn with the help of suitable sampling instrument (*see N-2*).

**N-4.2** Out of these portions, a small but equal quantity of material shall be taken and mixed thoroughly to form a composite sample of about 600 ml. The composite test sample shall be divided into three equal parts one for the purchaser, another for the supplier and the third to be used as a referee sample.

**N-4.3** The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the  $n$  containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

**N-4.4** All the individual and composite samples shall be transferred to separate containers and shall be sealed and marked with full identification

particulars given under **N-1.10**.

**N-4.5** The referee test samples consisting of a composite sample and a set of  $n$  individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute between the two.

#### N-5 NUMBER OF TESTS

**N-5.1** Tests for determination of freezing point and assay shall be conducted on each of the individual samples (*see N-4.3*).

**N-5.2** Tests for determination of remaining characteristics shall be conducted on the composite sample (*see N-4.2*).

#### N-6 CRITERIA FOR CONFORMITY

**N-6.1** The lot shall be declared as conforming to the requirements of this specification if the following conditions are satisfied:

##### N-6.1.1 Individual Samples

For individual test results of freezing point and assay, the average ( $\bar{X}$ ) and range ( $R$ ) shall be calculated and shall satisfy the expression  $\bar{X} - 0.6 R \geq$  the minimum specified value.

##### N-6.1.2 Composite Samples

The composite sample shall conform to the remaining requirements given in 3 and Table 1.

NOTE — Suitable sampling procedure may be used following the safety parameter.

**Table 5 Scale of Sampling**  
(Clause N-3.2)

Sl No.	Lot size	No. of Containers to be Selected
(1)	$N$ (2)	$n$ (3)
i)	Up to 50	3
ii)	51 to 100	4
iii)	101 to 150	5
iv)	151 and above	7

NOTE — In case of very small lots where the selection of three containers may be uneconomical, the number of containers to be selected and the method of judging the conformity of the lot to the requirements of specification shall be as agreed to between the purchaser and the supplier.

## ANNEX P

(Foreword)

## COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

<i>Organization</i>	<i>Representative(s)</i>
National Chemical Laboratory (NCL), Pune	DR C. V. RODE ( <b>Chairperson</b> )
Alkyl Amines Chemicals Ltd, Mumbai	SHRI S. V. NIKUMBHE SHRI SAMEER KATDARE ( <i>Alternate</i> )
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	SHRI K. L. RAPHAEL SHRI KIRTI GAJJAR ( <i>Alternate</i> )
All India Distillers Association (AIDA), New Delhi	SHRI V. N. RAINA
BASF India Ltd, Mumbai	SHRI KIRAN BHAT SHRI HEMAL ( <i>Alternate</i> )
CSIR-Central Drug Research Institute (CDRI), Lucknow	DR SANJEEV KANOJIYA
Chemical and Petrochemicals Manufacturers Association (CPMA), New Delhi	SHRI UDAY CHAND
Deepak Fertilizer, New Delhi	DR L. B. YADAWA SHRI SURESH AMLE ( <i>Alternate</i> )
Deepak Phenolics Ltd, Vadodara	SHRI DHARMESH SIDDHAPURIA SHRI SANDIP KUMAR PANDYA ( <i>Alternate</i> )
Dow Chemical International Pvt Ltd, Mumbai	SHRI V. MOHANDOSS SHRI GOVIND GUPTA ( <i>Alternate</i> )
Godavari Biorefineries, Mumbai	SHRI SHANUL LAXMANRAO PAGAR SHRI APPASAHEB J. WANI ( <i>Alternate</i> )
Gujarat Narmada Valley Fertilizers Company Ltd, Ahmedabad	DR R. M. PATEL SHRI C. S. PATEL ( <i>Alternate</i> )
Hindustan Organic Chemicals Ltd (HOCL), Mumbai	SHRI DELEEP KUMAR. K. SHRI PRADEESH NARAYANAN ( <i>Alternate</i> )
INEOS Styrolution India Ltd, Vadodara	DR KANAK DASS
India Glycols Ltd, Uttarakhand	DR R. K. SHARMA SHRI ALOK SINGHAL ( <i>Alternate</i> )
Indian Chemical Council (ICC), Mumbai	DR MRITUNJAY CHAUBEY SHRI J. SEVAK ( <i>Alternate</i> )
Indian Oil Corporation Ltd, Panipat	DR Y. S. JHALA



<i>Organization</i>	<i>Representative(s)</i>
Jubilant Life Sciences Ltd, Noida	SHRI MANSUKH D. KANZARIYA
Laxmi Organic Industries, Mumbai	SHRI J. P. SURYAVANSHI DR VIJAY S. MISHRA ( <i>Alternate</i> )
Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizers, New Delhi	SHRI O. P. SHARMA SHRI VARUN SINGH POONIA ( <i>Alternate</i> )
National Chemical Laboratory (NCL), Pune	DR RAVINDAR KONTHAM DR UDAYA KIRAN MARELLI ( <i>Alternate</i> )
Reliance Industries Ltd (RIL), Mumbai	SHRI SREERAMACHANDRAN KARTHA SHRI VASANT WARKE ( <i>Alternate</i> )
United Phosphorus Ltd (UPL), Mumbai	SHRI M. D. VACHHANI
In Personal Capacity (37 Nandanvan Society, Near GNFC Township, Narmadanagar, Bharuch – 392015)	DR MAYUR J. KAPADIA
BIS Director General	SHRIMATI MEENAL PASSI, SCIENTIST ‘F’/SENIOR DIRECTOR AND HEAD (PETROLEUM, COAL AND RELATED PRODUCTS) [REPRESENTING DIRECTOR GENERAL ( <i>Ex-officio</i> )]

*Member Secretary*  
KUMARI ADITI CHOUDHARY  
SCIENTIST ‘B’/ASSISTANT DIRECTOR  
(PETROLEUM, COAL AND RELATED PRODUCTS), BIS

(Continue from second cover)

While modifying the test methods, considerable assistance have been drawn from the following ASTMs:

- a) ASTM D 5386 Standard Test Method for Color of Liquids Using Tristimulus Colorimetry;
- b) ASTM D 4052 Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter;
- c) ASTM D 1218 Standard Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids;
- d) ASTM D 2340 Standard Test Method for Peroxides in Styrene Monomer;
- e) ASTM D 2121 Standard Test Methods for Polymer Content of Styrene Monomer; and
- f) ASTM D 4590 Standard Test Method for Colorimetric Determination of p-tert-Butylcatechol in Styrene Monomer or AMS ( $\alpha$ -Methylstyrene) by Spectrophotometry.

The composition of the committee, responsible for the formulation of this standard listed in Annex P.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.



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